INDEX

Expt. No.	Name of Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
1.	Preparation of Hydronium dichlorobis (dimethyl glyoximato cobaltate (III) $H[Co(dmgH)_2Cl_2]$	1 to 3			
2.	Preparation: Bis (tetra ethyl ammonium) tetra chloro (Cobaltate (II) $(Et_4N)_2[CoCl_4]$	4 to 7			
3)	Preparation: Bis (tetra ethyl ammonium) tetra chloro cuprate (II) $(Et_4N)_2[CuCl_4]$	8 to 10			

INDEX

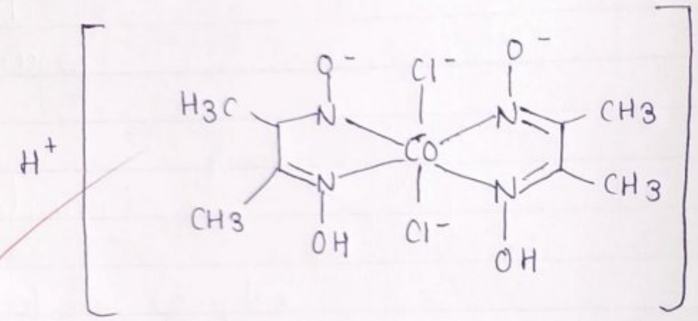
Expt. No.	Name of Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
4)	Preparation: Bis (ethylene diamine) Copper Sulphate $(Cu(en)_2]SO_4$.	11 to 14			
5)	Conductometry: Determination of electrolytic nature of inorganic compounds by conductance measurements.	15 to 18			
6)	Spectrophotometry: Determination of equilibrium of equilibrium constant by slope - intercept method for Fe^{3+}/SCN^- system.	19 to 23			

Reactions:

① $CoCl_2 + 2$ $CH_3-C=NOH$ / $CH_3-C=NOH$ $\xrightarrow{CH_3COCH_3}$ $H[Co(dmgH)_2]Cl_2$

② $Co^{2+} + Na_2C_{10}H_{14}O_8N_2 \longrightarrow CoC_{10}H_{14}O_8N_2 + 2Na^+$

Structure:



Observations:-

[I] Yield of the complex:

Beaker A = 3 g of $CoCl_2 \cdot 6H_2O$ + small amount of acetone
Beaker B = 3.3 g of $dmgH_2$ + small amount of acetone

Observed Yield of the complex = x = 1.2 gm.

II] Characterisation of the complex:

i) Solution in burette: 0.02 M EDTA solution.
ii) Solution in conical Flask: 10 cm^3 diluted solution + one test tube distilled water + 3 drops of 0.5 % xylenol orange + 2 N H_2SO_4 (red to yellow) + pH 6 by adding hexamine + warm about 40°C

Preparation of Hydronium dichloro bis (dimethylglyoxmato) cobaltate (III)

EXPERIMENT: No. 1 — $H[Co(dmgH)_2Cl_2]$ — Date / / — Page 1.

Aim: To prepare and characterise Hydronium dichloro bis (dimethylglyoximato) cobaltate (III) $H[Co(dmgH)_2Cl_2]$

Requirements: $CoCl_2 \cdot 6H_2O$, Dimethylglyoxime ($dmgH_2$), dry acetone, boiling water bath, 100 cm^3 beaker, 150 cm^3 conical flask, watch glass, 0.0 M EDTA, 0.5 % xylenol orange indicator, hexamine powder, etc.

Theory: When solution of $CoCl_2 \cdot 6H_2O$ in minimum volume of dry acetone is mixed with a solution of dimethyloxime in dry acetone and gently boiled, deep green coloured crystals of the complex form on cooling.

Procedure:

[I] Preparation of the complex:

(1) Dissolve 3.0 gm of $CoCl_2 \cdot 6H_2O$ in minimum volume of dry acetone in 100 cm^3 clean & completely dried beaker and cover it with a watch glass.
(2) Weigh 3.3 gm of dimethylglyoxime ($dmgH_2$) on a clean dry watch glass and dissolve it in little more than minimum volume of dry acetone in 100 cm^3 beaker. If required heat it on a boiling water bath to dissolve it
(3) Transfer the solution of dimethylglyoxime (If required, filter through filter paper) in a solution of $CoCl_2 \cdot 6H_2O$ and mix it well.
(4) Place the beaker on a boiling water bath and allow.

Sulabh®

Teacher's Sign:

iii) Indicator = 0.5 % xylenol orange indicator.
iv) End point : Red to yellow colour.

Observation Table:

Level	Burette Reading (cm^3)			Constant Burette Reading (cm^3)
	I	II	III	
Initial	0.0	0.0	0.0	
Final	5.0	5.0	5.1	$y = \underline{5.0}\ cm^3$
Difference	5.0	5.0	5.1	

◉ Calculations :-

I] Yield of the Complex :-

A) Theoretical Yield of the complex.

237.94 g of $CoCl_2 \cdot 6H_2O$ gives 362.94 g of the complex
∴ 3.0 g of $CoCl_2 \cdot 6H_2O$ gives $= \frac{362.94 \times 3}{237.94}$
$= 4.57$ g of the complex
Solvent is highly volatile 80 % of yield is expected i.e 3.6 gm.

B) Observed yield of the complex.

$x = \underline{\underline{1.2}}$ g of the complex

(i) Percentage yield of the complex :
4.57 g of the complex = 100 % yield.
∴ x g of of the complex $= \frac{1.2 \times 100}{4.57} = 21.88 \times 1.2$
$= \underline{\underline{26.25\ \%}}$

EXPERIMENT: No. Date / / Page 2

the reaction mixture to boil gently until deep green crystals of the complex start forming.
(5) Allow the reaction mixture to cool in air and then collect the crystals by filtration under solution. Wash it with dry acetone and then dry in air.
(6) Weigh the product to find yield is(x gm)

(II) Characterisation of the complex:

Analysis of hydronium dichlorobis (dimethylglyoximato) Cabaltate (III): for the percentage of cobalt:-

(1) Weigh accurately 0.4 g of the complex on a watch glass and transfer it qualitatively in a 250 cm^3 clean dry beaker.
(2) Add about 1-2 cm^3 of conc. HCl to the weighed complex and heat it on a sand bath to decompose it.
(3) Cool the residue and extract it with 25-30 cm^3 of distilled water. Dilute it to 100 cm^3 in standard measuring flasks shake well.
(4) Pipette out 10 cm^3 of the diluted solution in a 150 cm^3 conical flask and add one test tube of distilled water to it.
(5) Add 3 drops of 0.5 % xylenol orange indicator solution followed by very dilute or 2N H_2SO_4 until the colour just changes from red to yellow colour.
(6) Adjust the pH of solution to about 6 by adding powdered hexamine (red colour is restored).
(7) Warm the solution to about 40°C and titration it

Teacher's Sign:

EXPERIMENT: No. _____ Date / / Page 3

against 0.02 M EDTA solution from the burette with constant burette shaking till the colour changes from red to yellow colour (y cm^3)

Results:-

(1) Yield of the complex:

(A) Theoretical = 3.6 g

(B) Observed $(w_2 - w_1) = x$ = 1.2 g

(2) Percentage of copper present in the complex.

(A) Theoretical = 16.23 %

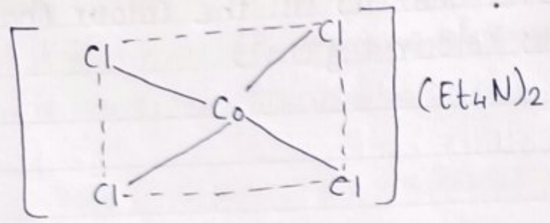
(B) Observed = 14.73 %

Reactions:

1) $CoCl_2 + 2Et_4NCl \longrightarrow (Et_4N)_2[CoCl_4]$

2) $Co^{2+} + Na_2C_{10}H_{14}O_8N_2 \longrightarrow CoC_{10}H_{14}O_8N_2 + 2Na^+$

Structure:



Observations:

(1) Preparation of the complex:

Beaker A: 1.2 g of $CoCl_2 \cdot 6H_2O$ + 5 cm^3 absolute alcohol

Beaker B: 1.9 g of tetraethyl ammonium chloride salt + 5 cm^3 absolute alcohol

Density of tetraethyl ammonium chloride = 1.08 g cm^{-3}

mass = 1.9 gm $\therefore$ volume = $\frac{\text{mass}}{\text{density}} = \frac{1.89}{1.08} = 1.75\ cm^3$

1) Weight of empty crucible = w_1 = 64.7 gm.
2) Weight of crucible + complex = w_2 = 65.3 gm
3) Weight of the complex = $w_2 - w_1$ = 0.6 gm

4) Observed yield of the complex = _____ gm.

EXPERIMENT: No. 2 — Preparation of Bis (tetraethyl ammonium) tetrachloro cobaltate(II) $(Et_4N)_2[CoCl_4]$ — Date / / Page 4

Aim: To prepare and characterise Bis (tetraethyl ammonium) tetrachloro cobaltate (II)

Requirements: $CoCl_2 \cdot 6H_2O$, tetraethyl ammonium chloride salt, absolute alcohol, conical flasks, sintered glass crucible, 0.5 % xylenol orange indicator, 0.02 M EDTA solution, watch glass, hexamine powder, standard measuring flask, waterbath, cotton plug, beakers, pipeclay triangle, 2N H_2SO_4

Theory: When $CoCl_2 \cdot 6H_2O$ solution in absolute alcohol is mixed with tetraethyl ammonium chloride solution in absolute alcohol and boiled on steam bath for a minute on cooling coloured crystals of tetraethyl ammonium complex compound. Since reaction is like non aqueous reaction; completely dried apparatus are required to be used.

Procedure:

[I] Preparation of the complex $(Et_4N)_2[CoCl_4]$:

(1) Weigh exactly 1.2 g of $CoCl_2 \cdot 6H_2O$ on a clean dry watch glass and transfer it in a dry 150 cm^3 conical flask labelled as A.

(2) Weigh accurately 1.9 gm of tetraethyl ammonium chloride salt ($Et_4NCl \cdot H_2O$) on a clean dry watch glass and transfer it gently to clean dry 150 cm^3 conical flask labelled as B.

Sulabh — Teacher's Sign:

Calculations :-

1) Yield of the complex :-

A) Theoretical yield of the complex.

237.94 g of $CoCl_2 \cdot 6H_2O$ = 460.94 g of the complex

∴ 1.2 g of $CoCl_2 \cdot 6H_2O$ = $\frac{460.94 \times 1.2}{237.94}$

= 2.325 g of the complex.

B) Observed yield of the complex = $w_2 - w_1$

= 65.3 − 64.7

= x g

= 0.6 g

8) Wash the conical flask and crystals with 1-2 cm^3 of absolute alcohol. Dry the crystals in air.

9) Collect the filtrate i.e mother liquor and evaporate it to recover maximum product.

10) Weigh the crucible with product (w_2 g) and find out yield of the product (x g)

(II) Characterisation of the Complex $(Et_4N)_2[CoCl_4]$:

Analysis of Bis (tetraethyl ammonium) tetrachloro cobaltate · $(Et_4N)_2[CoCl_4]$ for the percentage of cobalt:

1) Weigh accurately 0.400 g of the complex on a watch glass and transfer to a 250 cm^3 clean beaker.

2) Add 5-7 cm^3 conc. HNO_3 and heat it on a sand bath

Sulabh®

Teacher's Sign:

II] Analysis of the Complex for the percentage of Cobalt.

A] Theoretical percentage of cobalt:-

460.94 g of $(Et_4N)_2[CoCl_4]$ contains 58.94 g of cobalt

$\therefore$ 0.400 g of $(Et_4N)_2[CoCl_4]$ contains $= \frac{58.94 \times 0.400}{460.94}$

$= 0.0513$ g of cobalt

Now. 0.400 g of the complex contains 0.0513 g of cobalt

$\therefore$ 100 g complex contains $= \frac{0.0513 \times 100}{0.400}$

$= 12.83$ % of cobalt

(B) Observed percentage of cobalt:-

10 cm^3 of diluted solution required 2.5 cm^3 of 0.02 M EDTA

$\therefore$ 100 cm^3 of diluted solution required 10×2.5 cm^3 of 0.02 M EDTA

1000 cm^3 of 1 M EDTA = 58.94 g of cobalt

$\therefore 10 \times$ cm^3 of 0.02 EDTA $= \frac{58.94 \times 0.02 \times 10 \times 2.5}{1000}$

$=$ 0.0294 g of cobalt.

Now. 0.400 g of $(Et_4N)_2[CoCl_4]$ contains 0.0294 of cobalt.

$\therefore$ 100 g of $(Et_4N)_2[CoCl_4]$ contains $= \frac{0.0294 \times 100}{400}$

$=$

$=$ 7.36 % of cobalt

EXPERIMENT: No.

Date / /
Page 6

Covering beaker partially with a watch glass. Heat it nearly to dryness.

3) Extract the residue with 20-25 cm^3 of distilled water and boil it for 5 minutes.

4) Cool the solution and transfer it in a 100 cm^3 standard measuring flask. Wash the beaker 2-3 times with 5-7 cm^3 of distilled water each time and transfer the washings also.

5) Dilute the solution to 100 cm^3 with distilled water and shake it well.

6) Pipette out 10 cm^3 of the diluted solution in clean 150 cm^3 conical flask and add one test tube of distilled water to it.

7) Add 3 drops of 0.5 % xylenol orange indicator solution followed by very dilute or 2N H_2SO_4 until the colour just changes from red to yellow colour.

8) Adjust the pH of the solution to about 6 by adding powdered hexamine (red colour is restored)

9) Warm the solution to about 40°C and titration it against 0.02 M EDTA solution from the burette with constant shaking till the colour changes from red to yellow colour (4 cm^3)

Sulabh

Teacher's Sign:

(1)

(2)

Sulabh®

EXPERIMENT: No. 3

Preparation of Bis (tetraethyl ammonium) tetrachloro cuparoate (II) $(Et_4N)_2[CuCl_4]$

Date / /
Page 8

Aim : To prepare and characterise Bis (tetraethyl ammonium) tetrachloro cuprate (II)

Requirements : Copper chloride ($CuCl_2 \cdot 2H_2O$), tetraethyl ammonium chloride salt ($Et_4NCl \cdot H_2O$), absolute ethyl alcohol, crucible, 0.5 % fast salphone Black F indicator, 0.02 M EDTA solution, watch glass, 100 cm^3 standard measuring flask, conical flasks, pipeclay triangle, waterbath, cotton plug, beakers

Theory : When $CuCl_2 \cdot 2H_2O$ solution in absolute alcohol is mixed with tetraethyl ammonium chloride solution in absolute alcohol and boiled on a steam bath for a minute, on cooling gives yellow coloured crystals of bis (tetraethyl ammonium) tetrachloro cuparate complex compound. Since reaction is like non aqueous reaction, completely dried apparatus are required to be used.

Procedure :

[I] Preparation of the complex, $(Et_4N)_2[CuCl_4]$:

(1) Weigh exactly 1.0 g of copper chloride ($CuCl_2 \cdot 2H_2O$) on an absolutely dry watch glass and transfer it in a dry 150 cm^3 conical flask labelled as A.

2) Weigh accurately 2.0 g of tetraethyl ammonium chloride salt ($Et_4NCl \cdot H_2O$) on a dry watch glass and transfer it analytically in dry 150 cm^3 conical flask and label it as B.

Observation Table.

Level	Burette Reading			C.B.R (cm³)
	I	II	III	
Initial	0.0	0.0	0.0	
Final	6.0	6.1	6.0	4 = 6.0 cm3
Difference	6.0	6.1	6.0	

* Calculations:

(I) Yield of the Complex (Theoretical)

170.54 g of $CuCl_2 \cdot 2H_2O$ = 465.54 g of complex

1 g of $CuCl_2 \cdot 2H_2O$ = $\frac{465.54}{170.54}$

= 2.730 g of the complex

Observed yield of complex = $w_2 - w_1$ = x gm

= 0.6 gm

[II] Analysis of the Complex for the percentage of copper.

(A) Theoretical percentage of copper:

465.54 g of $(Et_4N)_2[CuCl_4]$ contains 63.54 g of copper.

∴ 0.400 gm of $(Et_4N)_2[CuCl_4]$ contains = $\frac{63.54 \times 0.400}{465.54}$

= 0.0546 g of copper.

Now 0.400 g of the complex contains 0.0546 g of copper.

∴ 100 g of complex contains = $\frac{0.0546 \times 100}{0.400}$

= 13.65 % of Copper

EXPERIMENT: No. Date / / Page 9

3) Very carefully add 6 cm^3 of absolute alcohol in each of the flask labelled as A and B.
4) Shake gently both the flasks to dissolve salts in each completely.
5) Now mix the solution in conical flask A with a solution in conical flask B slowly with shaking gently and cover the conical flasks with cotton plug.
6) Boil water in a water bath and put a pipe clay triangle in it. Boil the solution mixture in a conical flask in steam for a minute.
7) Cool the solution in air and collect the crystals on previously weighed sintered glass crucible (w_1 g) by filtration of the solution under suction.
8) Wash the conical flask and crystals with 1-2 cm^3 of absolute alcohol. Dry the crystals in air.
9) Collect the filtrate, i.e mother liquor and evaporate it to recover maximum product.
10) Weigh the crucible with product (w_2 g) and find out yield of the product (x g)

[II] Characterisation of the Complex $(Et_4N_2)[CuCl_4]$

Analysis of Bis (tetraethyl ammonium) tetrachloro cuparate $(Et_4N)_2[CuCl_4]$ for the percentage of copper.

1) Weigh accurately 0.400 g of the complex on watch glass and transfer it to a 250 cm^3 clean beaker.
2) Add 3.5 cm^3 of conc. HNO_3 and heat it on a sand bath

Teacher's Sign:

(B) Observed Percentage of Copper:

10 cm^3 of the diluted solution required cm^3 of 0.01 M EDTA solution.

∴ 100 cm^3 of the diluted solution required 10 × 6 cm^3 of 0.01 M EDTA solution.

1000 cm^3 of 1M EDTA = 63.54 g of copper.

∴ 10 × 6 cm^3 of 0.02 M EDTA $= \frac{63.54 \times 0.02 \times 10 \times 6}{1000}$

$= \frac{76.248}{1000}$

$= 0.07624$ g of copper

Now 0.400 g of $(Et_4N)_2[CuCl_4]$ contains 0.07624 g of Copper.

∴ 100 g of $(Et_4N)_2[CuCl_4]$ contains $= \frac{0.07624 \times 100}{0.400}$

$= \frac{7.6248}{0.400}$

$= 19.062$ % of Copper

EXPERIMENT: No. Date / / Page 10

covering beaker partially with watch glass. Heat it nearly to dryness

3) Extract the residue with 20-25 cm^3 of distilled water and boil it for 5 minutes.
4) Cool the solution and transfer in a 100 cm^3 standard measuring flask. wash the beaker 2-3 times with 5-7 cm^3 of distilled water each time and transfer the washings also.
5) Dilute the solutions in a clean standard measuring flask to 100 cm^3 with distilled water and shake it well.
6) Pipette out 10 cm^3 of the diluted solution in 150 cm^3 conical flask. Add 25 cm^3 or one test tube distilled water to it and shake well.
7) Add 5-7 cm^3 liq NH_3 and 4-5 drops 0.5 % Fast sulphone black F indicator to it.
8) Titrate it against 0.01 M EDTA solution from the burette till the colour changes from blue to dark green colour (4 cm^3)

Results:

(1) Yield of the complex:
(A) Theoretical = 2.730 g
(B) Observed $(W_2 - W_1) = x =$ 0.6 g.

(2) Percentage of copper present in the complex
(A) Theoretical = 13.65 %
(B) Observed = 19.06 %

Sulabh® Teacher's Sign:

Reactions:

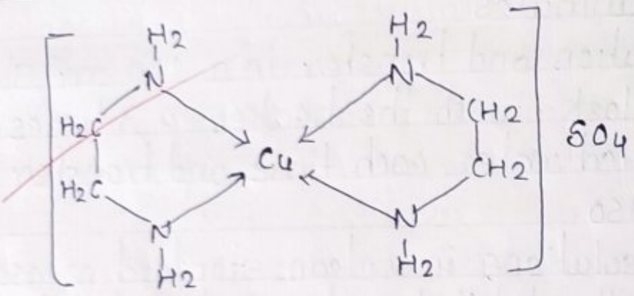
(1) $CuSO_4 + 2(C_2H_4(NH_2)_2 \longrightarrow Cu[C_2H_4(NH_2)_2]_2SO_4$

(2) $Cu^{2+} + NO_2C_{10}H_{14}O_8N_2 \longrightarrow CuC_{10}H_{14}O_8N_2 + 2Na^+$

(3) $C_2H_4(NH_2)_2 + 6H_2O \longrightarrow 2NH_4OH + 5H_2\uparrow + 2CO_2\uparrow$

(4) $NH_4OH + HCl \longrightarrow NH_4Cl + H_2O$

Structure:-



Observations:

[I] Preparation of the Complex

i) Solution in beaker : 2.5 g of $CuSO_4 \cdot 5H_2O$ + 5 cm^3 H_2O + 8-10 cm^3 80 % ethylene diamine + 15 cm^3 ethyl alcohol.

ii) Observed yield of the complex = ∞ gm = 3.9 gm

[II] Analysis of the complex for the percentage of copper.

i) Solution in Burette : 0.01 M EDTA

ii) Solution in conical flask : 10 cm^3 diluted solution + 1 test tube water + 5-7 liq NH_3 + 0.5 % Fast sulphone black F indicator

iii) Indicator : 0.5 % Fast sulphon black F indicator

iv) End point : blue to dark green.

EXPERIMENT: No. 4 — Preparation of Bis (ethylene diamine) Copper sulphate $[Cu(en)_2]SO_4$. Date / / Page 11

Aim: To prepare and characterise Bis (ethylene diamine) copper sulphate.

Requirements: Copper sulphate, 80 % ethylene diamine; Whatman filter paper No. 41, ethyl alcohol, 0.5 % Fast Sulphone black F indicator, 0.02 M EDTA solution, ice bath, beakers, watch glass, standard measuring flask conical flask, conc. HNO_3, sand bath, Liq NH_3, 1 % methyl orange indicator, 0.2 N HCl, pipette, Rtc.

Theory: Cu (II) reacts with ethylene diamine. It is a bidentate ligand, i.e it has two donor atoms like Nitrogen, which can donate a pair of electrons to form insoluble chelate compound. The coordination nuber of copper ion is four & can bind two bidentate ligands. When $CuSO_4 \cdot 5H_2O$ is dissolved in water, the octahedral $[Cu(H_2O)_6]^{2+}$ (d^9) ions are prevailing in aq. solution of Cu(II) salt. When ethylene diamine is added due to John-Teller effect, it undergoes tetragonal distortion producing distorted octahedral or square planar complex $[Cu(H_2O)_6]^{2+} + 2en \rightleftharpoons [Cu(en)_2]^{2+} + 6H_2O$. Reaction of $CuSO_4 \cdot 5H_2O$ with ethylene diamine gives $[Cu(en)_2]SO_4$

Procedure:

(I) Preparation of the complex:-

1. Dissolve 2.5 g of supplied quantity of $CuSO_4 \cdot 5H_2O$ in 5 cm^3 distilled water by warming in 50 cm^3 beaker.

Teacher's Sign:

Sulabh®

Observation table:

Reading level	Burette Reading I	II	III	C.B.R cm^3
Initial	0.0	0.0	0.0	
Final	13.5	13.5	13.6	$4 = 13.5\ cm^3$
Difference	13.5	13.5	13.6	

[III] Analysis of Bis (ethylene diamine) copper sulphate for the percentage of Ethylene diamine.

i) Solution in conical Flask : 0.15 g of complex + 25 cm^3 distilled water + 1-2 drops 1% methyl orange indicator.
ii) Solution in burette : 0.1 N HCl solution.
iii) Indicator : 1% methyl orange indicator.
iv) Endpoint : yellowish brown to bright orange.

Observation table:

Level	Burette Readings I	II	III	C.B.R (cm^3)
Initial	0.0	0.0	0.0	2
Final	28.9	28.9	29	$2 = 28.9\ cm^3$
Difference	28.9	28.9	29	

* Calculations:

(1) Yield of the Complex:

(A) Theoretical Yield of the complex

249.54 g of $CuSO_4 \cdot 5H_2O$ = 279.54 g of $Cu(en)_2SO_4$

$\therefore$ 2.5 g of $CuSO_4 \cdot 5H_2O = \frac{279.54 \times 2.5}{249.54}$

= 2.801 g of complex

EXPERIMENT: No. Date / / Page 12

2. Add 8-10 cm^3 80% ethylene diamine from the burette with constant stirring and stir for further 10 minutes.
3. Cool the solution in ice bath and add 10-15 cm^3 of ethyl alcohol to it.
4. Cool the solution and keep it aside for about 10-15 minutes.
5. Filter off the bright blue coloured precipitate through Whatman filter paper NO 41 using suction pump.
6. Wash the precipitate first with 1-2 cm^3 of ethyl alcohol.
7. Dry the suction pump for 15-20 min and then in air.
8. Weigh the product to find out yield (x g)

[II] Characterisation of the complex:

i) Analysis of Bis (ethylene diamine) copper sulphate for the percentage of copper.

1. Weigh accurately 0.400 g of the complex on watch glass and transfer to 250 cm^3 of beaker.
2. Add 5-7 cm^3 of conc HNO_3 and heat it on sand bath covering beaker partially with watch glass Heat it nearly to dryness
3. Extract the residue with 20-25 cm^3 of distilled water and boil it for 5 minutes.
4. Cool the solution and transfer it in 100 cm^3 standard measuring flask.
5. Dilute the solution in standard measuring flask

Sulabh®
Teacher's Sign:

(B) Observed yield of the complex = x g = 3.9 gm

[II] Analysis of the Complex for the percentage of copper:

(A) Theoretical percentage of copper:

279.54 g of Cu(en)2SO4 contain 63.54 g of copper

∴ 0.400 g of Cu(en)2SO4 contain = $\frac{63.54 \times 0.400}{279.54}$

= 0.091 g of copper.

Now 0.400 g of the complex contains 0.091 g of copper.

∴ 100 g of the complex contains = $\frac{0.091 \times 100}{0.400}$

= 22.78 % of copper.

(B) Observed percentage of copper:-

10 cm^3 of the diluted solution required 13.5 cm^3 of 0.01 M EDTA

∴ 100 cm^3 of the diluted solution required 10 × 13.5 cm^3 of 0.01 M EDTA

1000 cm^3 of 1 M EDTA = 63.54 g of copper.

∴ 10 × cm^3 of 0.01 M EDTA = $\frac{63.54 \times 10 \times 13.5 \times 0.01}{1000}$

= $\frac{85.7}{1000}$

= 0.0857 g of copper.

Now 0.400 g of Cu(en)2SO4 contains 0.0857 of copper

∴ 100 g of Cu(en)2SO4 contains = $\frac{0.0857 \times 100}{0.400}$

= $\frac{8.57}{0.400}$

= 21.6 % of copper.

EXPERIMENT: No. Date / / Page 13

to 100 cm^3 of distilled water and shake it well

6. Pipette out 10 cm^3 of the diluted solution in 150 cm^3 conical flask and add one test tube water to it.
7. Add 5-7 cm^3 Liq NH3 and 4-5 drops of 0.5 % Fast sulphone black Findicator to it.
8. Titrate it against 0.01 M EDTA solution from the burette til the colour changes from blue to dark green colour.
9. Repeat the titration to get constant readings (4 cm^3)

ii) Analysis of Bis (ethylene diamine) copper sulphate for the percentage of ethylene diamine.

10. Weigh accurately 0.150 g of the complex on a watch glass and transfer quantitatively in a 150 cm^3 conical flask. Add 25 cm^3 of distilled water to it and shake well.
11. After the complex dissolve and bluish turbid solution is obtained, add 1-2 drops of 1% methyl orange indicator to it. Dirty yellowish brown solutions forms.
12. Titrate it against 0.1 N HCl solution from the burette with constant shaking. The colour change from yellowish brown to greenish yellow to pinkish orange and finally to bright orange.
13. Repeat the above producer for two or more times to get a constant reading (7 cm^3)

Sulabh®

Teacher's Sign:

Results :-

(1) Yield of the complex :

(A) Theoretical = 2.801 g

(B) observed = (x) = 3.9 g

(2) Percentage of Copper present in the complex

(A) Theoretical = 22.73 %

(B) Observed = 21.6 %

(3) Percentage of Ethylene diamine present in the Complex :

(A) Theoretical = 42.92 %

(B) Observed = 57.8 %

Gulabh®

Teacher's Sign:

Observations:-

1) Room temperature = 25°C
2) Specific conductance of 0.1N KCl at 25°C = 0.01288
3) Observed conductance of 0.1N KCl at 25°C = 0.01324
4) cell constant = $\frac{\text{specific conductance}}{\text{observed conductance}}$

$= \frac{0.01288}{0.01324}$

$= 0.97$

5) Conductance of water at 25°C = 0.17×10^{-3} S

Observation table: For KCl

conc. of solution	observe conductance	Corrected conductance = obs. condn − condn of H_2O	specific conductance = corrected condn × cell constant	Molar conductance $\lambda m = \frac{K \times 1000}{C}$	$\sqrt{C}$
0.1	11.52×10^{-3}	11.35×10^{-3}	11.0095×10^{-3}	110.0	0.3162
0.05	6.32×10^{-3}	6.15×10^{-3}	5.9655×10^{-3}	119.3	0.2236
0.025	3.46×10^{-3}	3.29×10^{-3}	3.1913×10^{-3}	127.6	0.1581
0.0125	1.74×10^{-3}	1.57×10^{-3}	1.5229×10^{-3}	121.8	0.1118

For NaCl

conc. of solutions	Observe conductance	corrected conductance = obs. condn − conductance of H_2O	specific conductance = corrected conductance × cell constant	molar conductance $\lambda m = \frac{K \times 1000}{C}$	$\sqrt{C}$
0.1	8.50×10^{-3}	8.33×10^{-3}	8.080×10^{-3}	80.8	0.3162
0.05	4.90×10^{-3}	4.73×10^{-3}	4.588×10^{-3}	91.7	0.2236
0.025	2.84×10^{-3}	2.67×10^{-3}	2.589×10^{-3}	103.5	0.1581
0.0125	1.76×10^{-3}	1.59×10^{-3}	1.5423×10^{-3}	123.3	0.118

Determination of electrolytic nature of inorganic compounds by conductance measurements

EXPERIMENT: No. 5 Date / / Page 15

Aim:- Determination of electrolyte nature of given inorganic compound by conductance measurements:

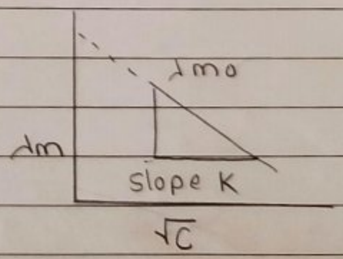
Requirements: 0.1 M solution of reagent A and reagent B conductivity water. 100 ml and 50 ml standard measuring flasks.

Theory: Conductance refers to the material to carry charge. The charge is carried by either electrons or ion cation & anions aqueous solution of salts cation free ions & hence can conduct electricity such salts are called electrolyte.

$$\lambda m = \frac{K \times 1000}{C}$$

where λm is molar conductance
K is specific conductance
C is concentration.

Where graph of λm vs $\sqrt{C}$ is plotted it gives stright line with slope K when line is extraplotted to $\sqrt{C} = 0$ it gives value of λm°



Calculation For KCl

1) $M_1V_1 = M_2V_2$
$(KCl) = (H_2O)$
$1 \times V_1 = 0.1 \times 50$
$V_1 = \frac{0.1 \times 50}{1}$
$V_1 = \underline{5}$

2] $M_1V_1 = M_2V_2$
$(KCl) = (H_2O)$
$1 \times V_1 = 0.05 \times 50$
$V_1 = \frac{0.05 \times 50}{1}$
$V_1 = \underline{2.5}$

3] $M_1V_1 = M_2V_2$
$(KCl) = (H_2O)$
$1 \times V_1 = 0.025 \times 50$
$V_1 = \frac{0.025 \times 50}{1}$
$V_1 = \underline{1.25}$

4] $M_1V_1 = M_2V_2$
$(KCl) = (H_2O)$
$1 \times V_1 = 0.0125 \times 50$
$V_1 = \frac{0.0125 \times 50}{1}$
$V_1 = \underline{0.625}$

① Corrected conductance = observed conductance − conductance of water

i) Corrected condⁿ = $11.52 \times 10^{-3} - 0.17 \times 10^{-3} = 11.35 \times 10^{-3}$
ii) Corrected condⁿ = $6.32 \times 10^{-3} - 0.17 \times 10^{-3} = 6.15 \times 10^{-3}$
iii) Corrected condⁿ = $3.46 \times 10^{-3} - 0.17 \times 10^{-3} = 3.29 \times 10^{-3}$
iv) Corrected condⁿ = $1.74 \times 10^{-3} - 0.17 \times 10^{-3} = 1.57 \times 10^{-3}$
v) ~~Corrected condⁿ =~~

② Specific conductance = corrected conductance x cell constant

① $K_1 = 11.35 \times 10^{-3} \times 0.97 = 11.0059 \times 10^{-3}$
② $K_2 = 6.15 \times 10^{-3} \times 0.97 = 5.9655 \times 10^{-3}$
③ $K_3 = 3.29 \times 10^{-3} \times 0.97 = 3.1913 \times 10^{-3}$
④ $K_4 = 1.97 \times 10^{-3} \times 0.97 = 1.5229 \times 10^{-3}$

③ $\sqrt{C}$

① $\sqrt{0.1} = 0.3162$
② $\sqrt{0.05} = 0.2236$
③ $\sqrt{0.025} = 0.1581$
④ $\sqrt{0.0125} = 0.1118$

EXPERIMENT: No. Date / / Page 16

Procedure:

1) Prepare 100 cm^3 of 0.1 M, 0.05 M, 0.025 M and 0.0125 M solution from the given sample of 1 M solution.
2) Pipette out 5, 2.5, 1.25, 0.625 cm^3 of 1 M solution reagent A in four 100 cm^3 standard measuring flask. Diluted each solution up to the mark with distilled water.
3) Take 50 cm^3 of 0.1 N KCl and 50 cm^3 distilled water in stoppered bottle.
4)

Teacher's Sign:

Sulabh

* Scale *

On X axis = 1 cm = 0.025 units

On Y axis = 1 cm = 0.5 units

Y-axis

150

140 — $(\Lambda m_2)_o$ = 140

130

120 — $(\Lambda m_1)_o$ = 118

Λ_m

110

100

90

80

50

0

0.1

0.2

0.3

0.4

X-axis

$\sqrt{C}$ →

KCl

NaCl

EXPERIMENT: No.

Date / /
Page 18

* Results:

1) Molar conductance of infinite dilution λ_m^∞ and electrolytic nature of KCl & NaCl

Compound	λ_m^∞	Nature of electrolyte
KCl	140	1:1
NaCl	118	1:1

Teacher's Sign:

EXPERIMENT: No. 6. Determination of equilibrium constant by slope-intercept method for Fe^{3+}/SCN^- system

Date / /
Page 19

Aim : Determination of equillibrium constant by slope intercept method for Fe^{3+}/SCN^- system

Requirements:

1) 0.002 M thiocynate solution : Dissolve 0.019 g of KSCN in minimum quality of distilled waters and dilute it to 100 cm^3 with 0.01 N HNO_3 in a standard measuring flask.
2) 0.1 M $Fe(NO_3)_3 \cdot 9H_2O$ solution : Dissolve 4.04 g of ferric nitrate in 0.5 N HNO_3 and dilute to 100 cm^3 with it in a standard measuring flask.
3) 256 cm^3 conc HNO_3 / dm^3 gives 4 N HNO_3.

To prepare.

i) 100 cm^3 of 0.01 N solution, take 0.064 cm^3 of conc HNO_3 / 100 cm^3
ii) 100 cm^3 of 0.5 N solution, take 3.2 cm^3 of conc HNO_3 / 100 cm^3
iii) 100 cm^3 of 2N solution, take 12.8 cm^3 of conc HNO_3 / 100 cm^3

- 50 cm^3, 100 cm^3 and 250 cm^3 beaker, spectrophotometer, 25 cm^3 pipette, 150 cm^3 conical flask, etc.

Theory: The study of complex formation involves two aspects (I) it's empirical formula (II) It's stability constant or equillibrium constant as its information involves reversible reaction.

① Empirical Formula.

It can be represent by three methods.

i) Job's continuous variation method.
ii) Mole ratio method
iii) Slope ratio method.

(II) Stability constant or equillibrium constant:

Stability constant of complex can be determine by constructing absorbance-concentration calibration plot, which allows the calculation of complex formed equation. The reaction of iron (III) with thiocynate, both of which are colourless, yield the orange red coloured product. iron (III) thiocynate complex ion as described by following equilibrium expression.

$$Fe^{3+} + SCN^- \rightleftharpoons Fe(SCN)_n^{2+}$$

$$\text{equillibrium constant}(K) = \frac{[Fe(SCN)]^{2+}}{[Fe^{3+}][SCN^-]} \quad \text{---} (1)$$

The absorbance can expressed as,

$$A = a\,b\,[Fe(SCN)]^{2+}$$

where a = absorptivity and b = path length.

The total molality of thiocyanate C_S and of iron C_F can be represented as:

$$C_S = [SCN^-] + [Fe(SCN)]^{2+}$$

$$C_F = [Fe^{3+}] + [Fe(SCN)]^{2+}$$

Putting the values of $[Fe(SCN)]^{2+} = \frac{A}{a \cdot b}$, $[Fe^{3+}] = C_F - \frac{A}{ab}$

and $[SCN^-] = C_S - \frac{A}{ab}$ in the eqn (1), we get

$$\frac{[Fe(SCN)]^{2+}}{[Fe^{3+}][SCN^-]} = \frac{\frac{A}{ab}}{C_F - \frac{A}{ab}\; C_S - \frac{A}{ab}}$$

By applying algebric eqn

$$\frac{C_F \times C_S}{A} = \frac{C_F + C_S}{ab} + \frac{1}{abK}$$

Gulabh®

Teacher's Sign:

This equation is in the form of $y = mx + c$ plot $C_F \cdot C_S$ against $(C_F + C_S)$

The straight line graph may have:

slope $= \frac{1}{ab}$ and intercept $= \frac{1}{abk}$

Hence, $K = \frac{\text{slope}}{\text{Intercept}}$

Procedure:

1) Pipette out 25 cm^3 of 0.002 M thiocynate solution carefully using rubber bulb in 250 cm^3 standard measuring flask. Add 60 cm^3 of 2N HNO_3 in it and dilute it upto the mark with distilled water will give 0.0002 M thiocynate solution.
2) Prepare 50 cm^3 of the following mixture of solutions in five searially numbered 50 cm^3 standard measuring flask.
3) Note down the absorbance (A) i.e optical density of each solution using 460 nm as λmax.

For graph :

I] $C_f \cdot C_s / A$

i) $\frac{0.004 \times 1.9 \times 10^{-4}}{0.30} = \frac{7.6 \times 10^{-7}}{0.30} = 2.53 \times 10^{-6}$

ii) $\frac{0.008 \times 1.84 \times 10^{-4}}{0.45} = \frac{1.472 \times 10^{-6}}{0.45} = 3.271 \times 10^{-6}$

iii) $\frac{0.012 \times 1.76 \times 10^{-4}}{0.49} = \frac{2.112 \times 10^{-6}}{0.49} = 4.310 \times 10^{-6}$

iv) $\frac{0.016 \times 1.68 \times 10^{-4}}{0.53} = \frac{2.624 \times 10^{-6}}{0.53} = 4.950 \times 10^{-6}$

v) $\frac{0.020 \times 1.60 \times 10^{-4}}{0.55} = \frac{3.2 \times 10^{-6}}{0.55} = 5.818 \times 10^{-6}$

[II] $C_f + C_s$

i) $0.004 + (1.9 \times 10^{-4}) = 4.19 \times 10^{-3}$
ii) $0.008 + (1.84 \times 10^{-4}) = 9.84 \times 10^{-3}$
iii) $0.012 + (1.76 \times 10^{-4}) = 13.76 \times 10^{-3}$
iv) $0.016 + (1.68 \times 10^{-4}) = 17.6 \times 10^{-3}$
v) $0.020 + (1.60 \times 10^{-4}) = 21.6 \times 10^{-3}$

* scale *

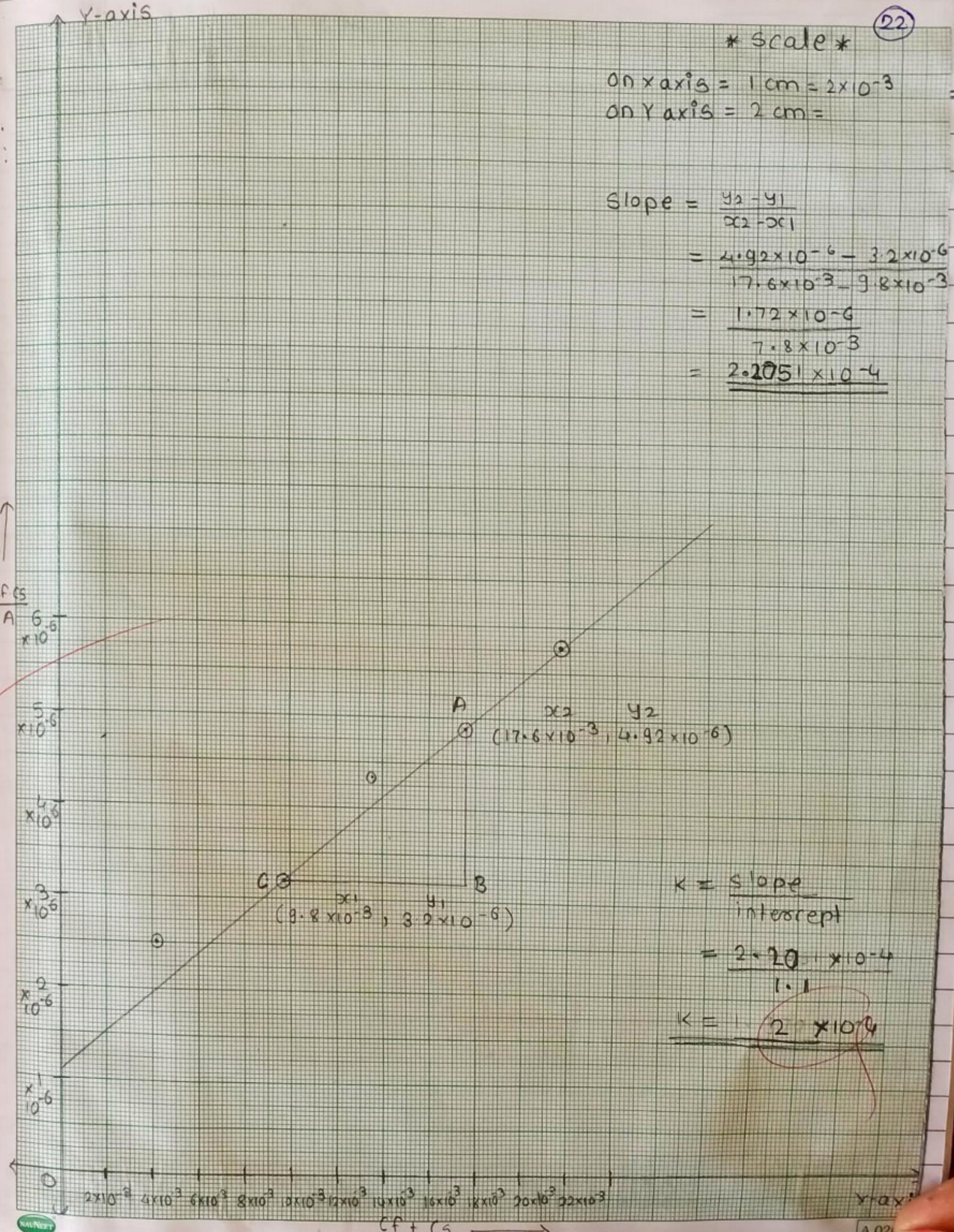
on x axis = 1 cm = 2×10^{-3}
on Y axis = 2 cm =

Slope = $\frac{y_2 - y_1}{x_2 - x_1}$

$= \frac{4.92 \times 10^{-6} - 3.2 \times 10^{-6}}{17.6 \times 10^{-3} - 9.8 \times 10^{-3}}$

$= \frac{1.72 \times 10^{-6}}{7.8 \times 10^{-3}}$

$= 2.2051 \times 10^{-4}$


Y-axis
$\frac{C_f C_s}{A}$
6×10^{-6}
5×10^{-6}
4×10^{-6}
3×10^{-6}
2×10^{-6}
1×10^{-6}
0
A
x_2 y_2
$(17.6 \times 10^{-3}, 4.92 \times 10^{-6})$
C
B
x_1 y_1
$(9.8 \times 10^{-3}, 3.2 \times 10^{-6})$
2×10^{-3} 4×10^{-3} 6×10^{-3} 8×10^{-3} 10×10^{-3} 12×10^{-3} 14×10^{-3} 16×10^{-3} 18×10^{-3} 20×10^{-3} 22×10^{-3}
$C_f + C_s$

$K = \frac{\text{slope}}{\text{intercept}}$

$= \frac{2.20 \times 10^{-4}}{1.1}$

$K = 2 \times 10^{-4}$

EXPERIMENT: No. _____ Date / / Page 23

* Results:

Equilibrium constant, $K = 2 \times 10^{-4}$